

ARL-RP-0594 APR 2017



The Performance of PS400 Subjected to Sliding Contact at Temperatures from 260 to 927°C

by Kevin C Radil and Chris DellaCorte

Reprinted from Tribology Transactions. 2016 Sep 14:1–8. doi: http://dx.doi.org/10.1080/10402004.2016.1231357.

Approved for public release; distribution is unlimited.

NOTICES

Disclaimers

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

Citation of manufacturer's or trade names does not constitute an official endorsement or approval of the use thereof.

Destroy this report when it is no longer needed. Do not return it to the originator.



The Performance of PS400 Subjected to Sliding Contact at Temperatures from 260 to 927°C

by Kevin C Radil

Vehicle Technology Directorate, ARL

Chris DellaCorte

National Air and Space Administration Glenn Research Center, Cleveland, OH

Reprinted from Tribology Transactions. 2016 Sep 14:1–8. doi: http://dx.doi.org/10.1080/10402004.2016.1231357.

REPORT D	Form Approved OMB No. 0704-0188					
data needed, and completing and reviewing the collection burden, to Department of Defense, Washington Head	ction information. Send comment lquarters Services, Directorate for ny other provision of law, no pers	s regarding this burden estin Information Operations and on shall be subject to any pe	ate or any other aspec Reports (0704-0188).	structions, searching existing data sources, gathering and maintaining the tt of this collection of information, including suggestions for reducing the 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. mply with a collection of information if it does not display a currently		
1. REPORT DATE (DD-MM-YYYY)	2. REPORT TYPE			3. DATES COVERED (From - To)		
April 2017	Reprint			June 2014–June 2015		
4. TITLE AND SUBTITLE	Ttop://iii			5a. CONTRACT NUMBER		
	icated to Sliding Co.	uras from	Sa. CONTINACT NOMBER			
The Performance of PS400 Subjected to Sliding Contact at Temper 260 to 927°C			ures from	5b. GRANT NUMBER		
				5c. PROGRAM ELEMENT NUMBER		
6. AUTHOR(S) Kevin C Radil and Chris DellaCorte				5d. PROJECT NUMBER		
				5e. TASK NUMBER		
				5f. WORK UNIT NUMBER		
7. PERFORMING ORGANIZATION NAM	E(S) AND ADDRESS(ES)			8. PERFORMING ORGANIZATION REPORT NUMBER		
US Army Research Laboratory						
ATTN: RDRL-VTP				ARL-RP-0594		
Aberdeen Proving Ground, MD 21005-5066						
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)				10. SPONSOR/MONITOR'S ACRONYM(S)		
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)		
12. DISTRIBUTION/AVAILABILITY STATEMENT						
Approved for public release; dis	stribution is unlimite	ed.				
13. SUPPLEMENTARY NOTES						
Reprinted from Tribology Trans	sactions. 2016 Sep 1	4:1–8. doi: http://	/dx.doi.org/10	0.1080/10402004.2016.1231357.		
14. ABSTRACT						
confronting today's engineers. In an patented, high-temperature solid lul calcium fluoride, PS400 is a varian dimensional stability. It was initiall high-temperature applications when	n attempt to provide a bricant coating. Made t of the earlier coating y developed to minimite e sliding friction and v	possible solution a from nickel-molyb , PS304, but is forn ize the start-stop w wear are a concern,	test program w denum-alumin nulated for higl ear in foil air b such as variab	ne of the most challenging tribological problems was initiated to evaluate PS400, a recently um, chrome oxide, silver, and barium fluoridener density, smoother surface texture, and greater earings but is expected to perform well in other le inlet guide vanes and process control valve the behavior of PS400 under reciprocating sliding		
contact at temperatures from 260 to against reciprocating PS400-coated For tests conducted below 927 °C,	o 927 °C. The tests were shaft specimens in a function coefficients rates ased at the lowest term	re performed on sta lat-on-cylinder cor nged from 0.37 to 0	tionary, uncoa figuration at H 0.84 with wear	ted cobalt-based superalloy bushings loaded fertz contact pressures from 14.1 to 20.1 MPa. factors on the order of 10 ⁻⁵ and 10 ⁻⁶ at the high covere limited because the coating was found to		
solid lubricants, friction, wear, high temperature						
sond lubricants, friction, wear, i	ingii temperature	47	40 NUMBER	10- NAME OF RECOONERS SERVICE		
16. SECURITY CLASSIFICATION OF:		17. LIMITATION OF	18. NUMBER OF	19a. NAME OF RESPONSIBLE PERSON Varian C. Radil		

Kevin C Radil

216-443-5047

19b. TELEPHONE NUMBER (Include area code)

PAGES

14

ABSTRACT

UU

b. ABSTRACT

Unclassified

a. REPORT

Unclassified

c. THIS PAGE

Unclassified



The Performance of PS400 Subjected to Sliding Contact at Temperatures from 260 to 927°C

Kevin Radil^a and Chris DellaCorte^b

^aArmy Research Lab–Vehicle Technology Directorate, Cleveland, OH, USA; ^bNASA Glenn Research Center, Cleveland, OH, USA

ABSTRACT

Adequate high-temperature lubrication between loaded surfaces in sliding contact can be one of the most challenging tribological problems confronting today's designers. In an attempt to provide a possible solution a test program was initiated to evaluate PS400, a recently patented, high-temperature solid lubricant coating. Made from nickel-molybdenum-aluminum, chrome oxide, silver, and barium fluoridecalcium fluoride, PS400 is a variant of the earlier coating, PS304, but is formulated for higher density, smoother surface texture, and greater dimensional stability. It was initially developed to minimize the start-stop wear in foil air bearings but is expected to perform well in other high-temperature applications where sliding friction and wear are a concern, such as variable inlet guide vanes and process control valve stems. To better define its operational capabilities, a series of tests was conducted to study the behavior of PS400 under reciprocating sliding contact at temperatures from 260 to 927°C. The tests were performed on stationary, uncoated cobalt-based superalloy bushings loaded against reciprocating PS400-coated shaft specimens in a flat-on-cylinder configuration at Hertz contact pressures from 14.1 to 20.1 MPa. For tests conducted below 927°C, friction coefficients ranged from 0.37 to 0.84 with wear factors on the order of 10^{-5} and 10^{-6} at the high temperatures but substantially increased at the lowest temperature. Data collected at 927°C were limited because the coating was found to be dimensionally unstable at this temperature.

ARTICLE HISTORY

Received 1 February 2016 Accepted 29 August 2016

KEYWORDS

Solid lubricants; friction; wear; high temperature

Introduction

High-temperature lubrication between loaded surfaces in sliding contact is an extremely challenging problem confronting today's designers. Due to oxidation, the upper limit of the best liquid lubricant is around 300°C. For higher temperatures polytetrafluoroethylene and molybdenum disulfide(MoS₂) are two very popular solid lubricants that produce adequate low friction at temperatures in the range of 250 to 400°C, beyond which they begin to lose their effectiveness (Miyoshi (1)). In an attempt to extend capabilities into higher temperature regimes, the National Aeronautics and Space Administration (NASA) began developing a family of solid lubricant coatings over 30 years ago to combat this deficiency (Sliney (2)). Designated as PS100, it was the first plasma-sprayed composite coating based on a ceramic matrix of glass and nickel-chromium (NiCr) infused with calcium fluoride and silver to provide friction and wear protection. Unfortunately, its softness resulted in wear rates that made it impractical for most applications. The next coating, PS200, addressed the high wear rates by replacing the glass with chrome carbide to improve its hardness and wear resistance (DellaCorte (3)). Unfortunately, it did not garner much interest because its hardness required expensive diamond grinding to achieve a proper finish. Building on the lessons learned from PS100 and PS200, a new family of coatings was

developed, motivated by the need for wear-resistant shaft coatings for foil air bearing applications. Designated as PS300, it combined NiCr as the binder and CrO2 as the hardener, resulting in a coating with moderate hardness while still retaining the silver and fluorides for low- and high-temperature lubrication capabilities (DellaCorte and Edmonds (4)). Further refinements in PS300 led to PS304, which had improved adhesion and tribological performance. Experiments in the lab on PS304 confirmed its lubricating effectiveness because it allowed tens of thousands of start-stop sliding cycles at high temperatures with minimal wear (DellaCorte, et al. (5)). The extensive lab testing also revealed that it suffered from dimensional instability and high surface roughness due to porosity. The dimensional instability stems from the metallic chromium precipitating in the matrix causing the coating to swell by up to 5% (DellaCorte, et al. (6); Lubell, et al. (7)). For coated parts to meet and retain their dimensional tolerance in a high-temperature environment, the coating first had to undergo heat treatment at 540°C for 150 h before any grinding process. Though this is an effective solution, the extra step adds to the coating's complexity and manufacturing costs. Additionally, the high surface roughness was linked to voids forming during the plasma spray process and possibly from soft fluoride material being pulled from the matrix by the grinding wheel.

The development of PS

The development of PS400 grew from the effort to address these major shortcomings, adding to the legacy of a family of high-temperature solid lubricant coatings capable of providing lubricant protection up to 760° C (DellaCorte and Edmonds (8)). By radically changing the coating's binder from NiCr-CrO₂ to NiMoAl while retaining Cr₂O₃ as the wear-resistant hardener and silver and barium fluoride–calcium fluorides as the lubricants, the coating improved on its dimensional stability sufficiently to eliminate the need for a postspray heat treatment. Furthermore, the substitution with NiMoAl, coupled with halving the amounts of fluoride and silver from 10 to 5%, produced a denser coating that resulted in a much improved surface finish without sacrificing performance.

As a shaft coating, PS304 allowed foil bearings to endure tens of thousands of high-temperature start-stop cycles without any significant wear to the bearing's top foils or coating when supporting loads up to 30 kPa. Limited results from start-stop tests on PS400 mirrored the same general trend of low friction and wear as PS304, a result not unexpected considering the constituent similarities between the two. Both coatings also exhibit the same wear behavior in sliding contact. When undergoing repeated high-temperature sliding cycles the coatings generate a smooth oxide glaze on the surface. This lubricious film is responsible for the low friction and wear results. The sliding also acts as a fine wear mechanism to polish the coating and bearing foils into conforming surfaces, which improves retention of the thin air film for increased performance. Additionally, the high number of sliding contact cycles promotes the transfer of solid lubricants to the other surface, forming a film that further reduces the coefficient of friction even though in foil bearings this sliding event only lasts for a relatively quick moment at startup and shutdown.

PS400's successful performance with foil bearings is not a true measure of its abilities or limitations. Given that adhesion tests performed from room temperature to 760°C measured the tensile strength in the range of 20-24 MPa, it is clear that foil bearing applications that typically support loads below 100 kPa represent nearly benign shear conditions (DellaCorte, et al. (6)). With the adhesion test failures being interstitial, it was important to explore higher shear limits of the coating and whether other high-temperature, higher load sliding contact applications could take advantage of PS400's lubricating capabilities. Such candidate applications include process control valve stems, inlet guide vane bushings, butterfly valve stems, and waste gate valves for turbochargers. Compared to foil bearings, these examples represent very different operating conditions; notably, the sliding surfaces remain in contact, which may hinder the development and growth of the oxide layer. It may also allow fine wear particles to collect. This is not an issue with foil bearings because the foil and shaft surfaces become separated by a thin air film after lift-off, thereby allowing a healthy oxide layer to re-form on any freshly exposed PS304 surfaces. In addition, a majority of the wear particles are displaced either from the constant air exchange due to the natural pumping action of the bearing or from the presence of axial cooling air. For surfaces constantly in contact without a similar air exchange mechanism, the possibility exists that wear particles could remain in the contact zone, resulting in an accelerated wear rate with increased severity. With an abrasive wear mechanism in place, the lubricious oxide layer would have a difficult time forming and re-forming because it would be constantly worn away as demonstrated by the thrust washer tests in Blanchet, et al. (9). However, other reports suggest that surfaces in constant sliding contact can still form the lubricious oxide layer (Striebing, et al. (10); Radil and DellaCorte (11)). There is also a successful commercial application of PS304 bushings lubricating furnace rollers at temperatures up to 520°C, so these concerns may be unwarranted (NASA (12)).

This article provides details on the dry sliding performance of NASA PS400 as a function of temperature and load. Cobalt alloy bushings were loaded against PS400-coated rods under reciprocating sliding contact at 1 Hz. The tests were conducted at various temperatures from 260 to 927°C and loads from 222.2–444.4 N, generating Hertz contact pressures from 14.1 to 20.1 MPa, respectively. Each test ran for 50,000 cycles. Torque output was collected throughout the entire test and used to calculate the static coefficient of friction. Upon disassembly, wear measurements on the bushing and PS400 were made using a stylus surface profilometer.

Test apparatus/procedure

Shown in Fig. 1 is the initial test hardware. The bushings were made from a cobalt-based superalloy with an inner diameter of 12.73 mm, outer diameter of 56.39 mm, and width of 12.7 mm. This material was chosen based on its common usage in high-temperature aerospace applications. The entire bushing's surface was polished with an average roughness of 0.302 μ m. Material hardness was measured to be HRC 39.

The rods are made from a nickel-based superalloy and are coated with PS400 at three different locations along its axial length to allow for three separate tests per rod. The substrate material was chosen based on its common usage in high-temperature aerospace applications. A flat at one of the ends (not shown) helped to secure the rod in place with a set screw. Surface preparation for plasma spraying the PS400 followed the directions in DellaCorte and Edmonds (8). The rod was coated and then ground to a final diameter of 12.67 mm, which provided a radial clearance of 30 μ m between the rod and the



Figure 1. Bushing and rod test specimens. The three dark bands on the two rods are PS400.

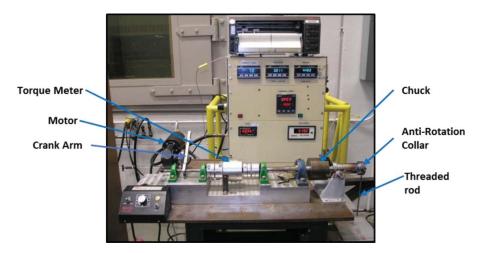


Figure 2. Reciprocating test rig.

bushing. The coating's average surface roughness after grinding was 1.129 μ m. The hardness of the coating was not measured but is given in Stanford (13) as approximately HRA 59.

The test rig is shown in Fig. 2. The rig consists of a 1/3 hp electric motor, crank arm, torque meter, a three-jaw chuck, rod holder (not shown), threaded rod, and antirotation collar. The rod holder is made from a 6-in.-long, 2.5-in. outer diameter round stock with an axial hole in the middle for placement of the rod. Three radial holes along the rod holder's length are provided for setscrews to hold the rod. The rod holder is secured by the three-jaw chuck and sits inside the antirotation collar. The antirotation collar secures the bushing and prevents it from rotating. The torque meter measures and sends the rotational friction data to the chart recorder. It also responsible for health monitoring during unattended operation and can stop the test if necessary. The bushing is loaded against PS400 by a threaded rod that extends from the bushing downward to a cable attached to the pneumatic load cylinder sitting below the test rig. A clam shell furnace for high-temperature testing (not shown) slides underneath and closes around the bushing.

To prepare the specimens for testing, they were first ultrasonically cleaned in methanol and then air dried. Once dry, the

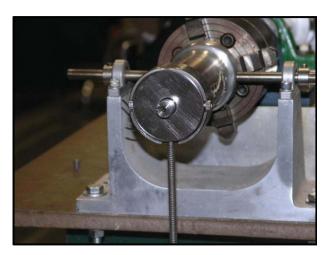


Figure 3. Test rig showing bushing in place on PS400-coated rod and secured in the antirotation collar. Threaded rod is part of the loading mechanism. Rod holder and furnace not shown.

PS400-coated rod was slid into the rod holder and the flat on the rod was aligned with one of the three radial holes. A stainless steel setscrew was fed into the hole and tightened against the flat on the rod. A second setscrew was then fed into the hole to lock in the first setscrew. The antirotation collar was installed over the holder while concurrently sliding the bushing onto the rod and into the end of the collar. This setup can be seen in Fig. 3. Once the bushing was secured to the collar with a couple of Allen head bolts the lower section of the furnace was slid below the bushing and the threaded rod was fed through a small hole in the furnace and screwed into the bushing. The cable attached to the pneumatic loader and load cell was then connected to the threaded rod. The upper section of the furnace was then lowered, thereby enclosing the bushing. At this point the furnace was activated and the bushing was heated to the desired temperature and allowed to soak for at least 30 min. Upon completion of the heating period, the motor was turned on and adjusted to produce a reciprocating speed of 1 Hz with a stroke angle of 15°, producing 30° of motion for one cycle. The load was applied and the chart recorder turned on to capture the resulting torque throughout the 50,000 cycles. The half-width of the torque trace represented the static coefficient of friction between PS400 and the cobalt bushing surface.

The test matrix consisted of subjecting the PS400 and bushing to three different loads, 222.4, 333.6, and 444.8 N, at temperatures of 260, 538, 760, and 927°C. The loads were chosen based on the operating limits of the test rig. To calculate the Hertz contact stress at each load and temperature, the elastic modulus for PS400 was determined by applying a polynomial curve fit to the data given in Standford, et al. (14). These data are for PS300 coating, but PS400's similar constituent amounts suggest that the values should be comparable, at least close enough to give reasonable estimates. PS300's average Poisson ratio of 0.28 at room temperature was also used. The resulting contact stresses are provided in Table 1.

Test results and discussion

After obtaining nonuniform wear scars on the bushing inner diameter and PS400 after the first few tests it was obvious that it was going to be difficult to achieve acceptable uniform

7 **(**) K.1

Table 1. Contact conditions, friction, and volumetric wear results for PS400 and cobalt bushing at various temperatures.

Temperature (°C)	Load (N)	Contact stress (MPa)	PS400 wear factor (mm ³ /N-m)	Cobalt wear factor (mm ³ /N-m)	Static coefficient of friction
760	222.4	14.1	9.45×10^{-6}	1.79×10^{-6}	0.43
760	333.6	17.2	1.18×10^{-5}	1.02×10^{-6}	0.40
760	444.8	19.9	1.10×10^{-5}	1.03×10^{-6}	0.37
538	222.4	14.2	3.00×10^{-6}	2.34×10^{-6}	0.41
538	333.6	17.3	3.69×10^{-6}	1.43×10^{-6}	0.40
538	444.8	20.0	3.81×10^{-6}	1.01×10^{-6}	0.36
260	222.4	14.2	1.87×10^{-4}	8.25×10^{-5}	0.82
260 ^a	333.6	17.4	1.23×10^{-3}	1.22×10^{-4}	0.84

^aTest performed for 2 h.

contact between the curved surfaces of the bushing inner diameter and the rod's outer diameter. This problem was alleviated by cutting the bushing's circular hole square using wire electric discharge machining (EDM) as shown in Fig. 4 to produce flaton-cylinder contact with the rod. This new configuration accommodated any slight misalignment between the rod and bushing while still retaining solid, uniform contact. The EDM process, however, resulted in a rougher surface finish, $R_a=3.868~\mu{\rm m}$, which is an order of magnitude higher than that of the original polished surface.

Posttest results for the PS400-coated rods and the cobalt bushing are shown in Figs. 5–10 and represent the highest loading condition tested for each temperature. Figures 5 and 6 illus-

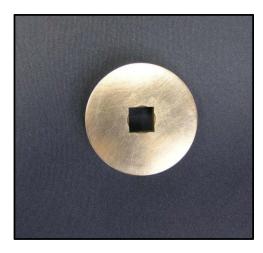


Figure 4. Modified cobalt bushing with square hole.



Figure 5. PS400 wear scar after testing at 760°C and 444.4 N. The scar exhibits a fully formed black oxide layer with light abrasive wear marks and polishing.

trate the PS400 and bushing wear scars produced during testing at 760°C and 444.4 N. As Fig. 5 shows, the PS400 wear scar exhibits a glossy black finish indicative of the oxide coating typically produced during sliding contact. The presence of a black finish demonstrates that an oxide layer is still able to form while in constant sliding contact with the bushing. Its rectangular shape also suggests mostly uniform load distribution between the specimens. Optical microscopy identified the presence of two wear mechanisms, polishing and light abrasion. The sliding marks are in the direction of motion, most likely from wear debris and/or the initial high surface roughness of the EDM bushing surface rubbing against a softer PS400 surface. Polishing was mainly evident between the sliding marks.

The wear scar on the bushing in Fig. 6 has characteristics similar to the PS400 wear scar with sliding marks and areas of polishing. Its black finish suggests that material transfer occurred from PS400 to the bushing. To check, an energy-dispersive spectroscopy (EDS) X-ray was performed on the wear scar and the results are shown in Fig. 7. The analysis confirmed the presence of all of PS400's matrix and lubricant phases on the bushing's surface, especially the high-temperature solid lubricants BaF_2/CaF_2 , which are pertinent to achieving low friction and wear.

Shown in Figs. 8 and 9 are the wear scars of the PS400 coating and bushing, respectively, after operating at 538°C and 444.4 N. The PS400 wear scar in Fig. 8 is very similar to the wear scar in Fig. 5, complete with the presence of the black oxide coating. Wear is uniform, with wear marks in the direction of sliding from abrasion coupled with areas of polishing.

The wear scar on the bushing in Fig. 9 is also similar to the bushing wear scar in Fig. 6 with sliding marks and areas of

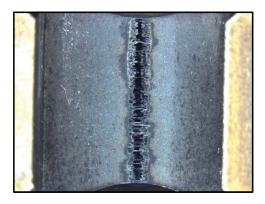


Figure 6. Bushing wear scar after testing at 760°C and 444.4 N. The wear scar exhibits light abrasive wear marks and polishing. The black film indicates a transfer of fluoride lubricants from the PS400.

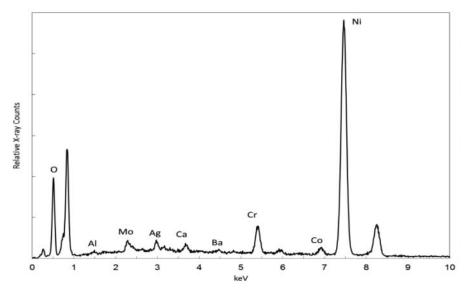


Figure 7. EDS X-ray spectrum of bushing after sliding against PS400-coated rod at 760°C.

polishing. The presence of a black finish in the wear scar indicates that there was a film transfer from PS400 to the bushing.

The results for the PS400 coating and bushing tests at 260° C and 333.6 N are shown in Figs. 10 and 11 and exhibit definite signs of severe abrasive wear. The test at 260° C and 222.4 N was run to completion but the excessive wear rate at 333.6 N caused the test duration to be shortened to 2 h (7,200 cycles) and cancellation of the subsequent test at 444.8 N.

Both wear scars demonstrate severe abrasive wear and are void of the black finish. The coating's performance at this temperature was not unexpected because PS400 is tailored for high-temperature environments, as the tests at 760 and 538°C demonstrated. An EDS analysis on the bushing wear scar, shown in Fig. 12, indicates that material transfer from PS400 to the bushing did occur, but 260°C is not high enough to activate the lubricating properties of the fluorides. Hence, the black lubricious oxide layer failed to form during sliding contact, leaving silver as the predominant solid lubricant to control the coating's friction and wear characteristics. Most likely silver's low concentration of 5% is insufficient to provide adequate lubrication at contact pressures present in this study, resulting in high wear.

An attempt was made to conduct tests at 927°C starting with a 222.4 N load. During the first hour of operation, the sliding

Figure 8. PS400 wear scar after testing at 538°C and 444.4 N. The scar exhibits a fully formed black oxide layer with light abrasive wear marks and polishing.

friction was observed to slowly increase, prompting stopping of the test as a precaution to check on the contact conformity between the two specimens. Once the specimens had cooled, a considerable amount of effort was needed to remove the bushing from the rod. Measurements with a micrometer indicated that the PS400 coating underwent a volumetric change, from either surface oxidation or an internal chemical reaction, causing the rod diameter to increase by a little over 20 μ m. To further investigate this phenomenon, the specimen was placed in a 927°C oven and allowed to soak for 24 h. Once cooled to room temperature, measurements confirmed that the rod diameter grew an additional 66 μ m for a total diametric growth of about 86 μ m, verifying that PS400 is not dimensionally stable at 927°C and above. The coating surface also exhibited a silvery finish, possibly signifying that a transformation had occurred. Therefore, no additional tests were conducted at this temperature. Further investigation into the cause of this behavior was not pursued because it is beyond the scope of this article.

The bushing and PS400 wear factors and friction data are shown in Table 1. The wear factor is determined by dividing the scar's wear volume by the load and the distance traveled. In

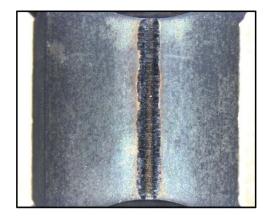


Figure 9. Bushing wear scar after testing at 538°C and 444.4 N. The wear scar exhibits light abrasive wear marks and polishing. The black film indicates a material transfer from the PS400.

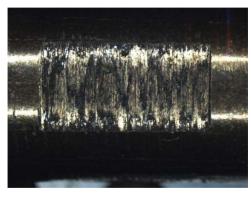


Figure 10. PS400 wear scar after testing for 2 h at 260°C and 333.6 N. The scar exhibits severe abrasive wear.

this case, with the rig reciprocating at 1 Hz and traveling 30° per cycle, 50,000 cycles translates to 166.25 m of distance traveled. The wear volume was estimated by first taking a trace along the scar's length with a stylus profilometer and using the profile to calculate the cross-sectional area of the removed material. An example of a length profile is shown in Fig. 13 and its shape is representative of the other resulting wear profiles. A second trace was made across the scar to obtain its width. Knowing the width and cross-sectional area allowed calculation of the wear volume. These measurements, however, relied on the subjective placement of horizontal and vertical lines in the profiles. For example, to obtain the wear depth, two horizontal lines were placed in the length profile, one representing the top surface of the coating or bushing and the other the bottom of the wear scar, each attempting to balance the peaks and valleys of the profile trace. The measured difference between the two was then taken as the wear depth. The same method was followed when determining the scar's length and width except that vertical lines were used.

According to the data in Table 1, PS400 and the cobalt bushing exhibit much lower wear factors at 538 and 760°C for all loads than at 260°C, a result not unexpected due to the presence of the lubricious oxide films on both the coating and bushing. The low wear results from the sliding contact being confined to the mating fluoride films and not between the substrate materials. Because measurements were not taken at intermittent points during the test it is unclear whether the wear on the PS400 or bushing occurred during initial break-in or throughout the test.



Figure 11. Bushing wear scar after testing for 2 h at 260°C and 333.6 N. The scar exhibits severe abrasive wear and is without the black lubricious transfer film.

However, the abrasive wear marks do suggest that wear continued on some level even after break-in and film formation, possibly from bushing surface roughness or wear debris. In addition, the lack of contact between substrate materials suggests that the coating may be capable of withstanding contact pressures higher than those produced in this study.

Comparing the wear factors for the tests at 538°C suggests a trend of increasing coating wear with a corresponding increase in load. The wear factors at 760°C follow this trend as well from 222.4 to 333.6 N but the wear drops slightly when the load is further increased to 444.8 N. This discrepancy is most likely a consequence of the subjective approach in collecting the wear volumes from the wear scar profiles. As stated previously, this approach estimates the amount of removed material and has a direct effect on the mantissa of the wear factor but little effect on the exponent. Hence, the mantissa values for the wear factors in Table 1 should also be considered as estimates, leaving the exponent to convey the level of performance. Furthermore, comparison of wear factors is typically deemed significant when an order of magnitude difference exists, especially when the data sampling size is small. Therefore, the wear factor exponents at 538 and 760°C suggest that the load had very little effect, at least for the given load range. Extending the range between the minimum and maximum loads would most likely provide a better understanding of the effects of load on PS400's wear performance.

To evaluate the accuracy of this subjective approach the wear scar area from Fig. 13 was determined using numerical integration. For depth measurements the original surface was taken to reside at a height of 15 μ m. Using the area measurement from numerical integration, the wear factor was found to be 1.32×10^{-5} mm³/N-m, which is a little more than the subjective result of 1.10×10^{-5} mm³/N-m. This is a 20% increase in the mantissa but, more important, the exponents are identical. Therefore, the subjective measurement approach provides reasonable estimates for the resulting wear factors.

It is interesting to note that there appears to be a legitimate order of magnitude jump in the PS400 wear factor for the three test loads when the ambient temperature was raised from 538 to 760°C. For example, the wear factor at 538°C and 333.6 N was 3.69×10^{-6} mm³/N-m but jumped to 1.18×10^{-5} mm³/N-m at 760°C. This result is counterintuitive because the fluorides should be providing the same level of protection at these two temperatures. An investigation into this phenomenon pointed to the maximum service temperature of the NiMoAl matrix material. Because PS400 is a composite coating, each constituent contributes, to a certain extent, to the physical makeup and behavior of the coating. In PS400, the matrix material is responsible for defining the coating's overall physical and mechanical properties, so the individual characteristics of NiMoAl are going to be the most influential. Based on the information supplied by the manufacturer, the recommended service temperature for NiMoAl is below 600°C, so it is logical to assume that this service temperature might apply to PS400 as well. Therefore, it is possible that exposing the coating to

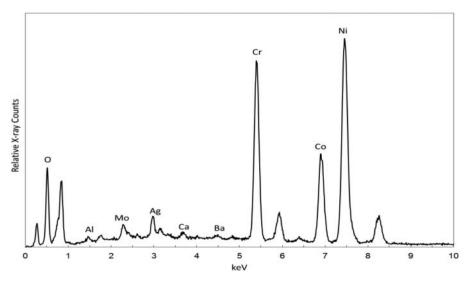


Figure 12. EDS X-ray spectrum of bushing wear scar after sliding against PS400-coated rod at 260°C.

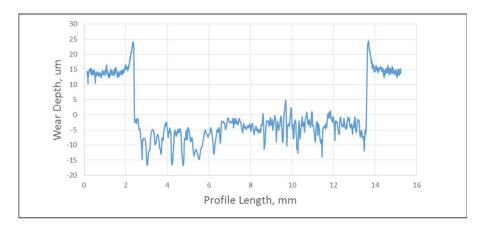


Figure 13. Profilometer profile of the PS400 wear scar from testing at760°C and 444.8 N.

 $760^{\circ}\mathrm{C}$ caused a sufficient softening of the matrix to allow the wear to increase.

The test results suggest PS400 is not a viable candidate as a wear resistance coating in applications where the temperature is constantly below 260°C and the contact pressures equal or exceed the loading conditions applied during this study. At 222.4 N the amount of wear was two orders of magnitude higher than the results at 760 and 538°C. This difference increases to three orders of magnitude at a load of 333.6 N and would probably be higher if the test was not stopped after undergoing only 7,200 cycles. As mentioned previously, the coating's low concentration of silver is most likely the reason for the high amounts of wear.

The static coefficient of friction was obtained by measuring the width of the recorded torque trace minus the residual torque in the rig. The residual torque represents the static drag force in the test rig's pillow block bearings and was determined for each load condition as shown in Table 2. As indicated by the test results in Table 1, the general trend is, for a constant load, that the coefficient of friction does not demonstrate any significant change as a function of

temperature from 538 to 760°C. Previously reported friction data from pin-on-plate testing in DellaCorte and Edmonds (8) are somewhat lower but they represent kinetic friction from tests using different material combinations and unidirectional motion. In addition, the hemispherical tips of the pins were polished smooth.

The friction data also demonstrate a general trend for temperatures at 760 and 538°C. As the load is increased for a constant temperature, the data suggest that the friction coefficient decreases. It is unclear why this occurs, but one possible explanation is that the higher loads promote a greater amount of PS400 to transfer from the rod to the bushing surface thereby reinforcing the PS400 in the PS400 sliding contact scenario.

Table 2. Measured residual rig torque as a function of applied load.

Load (N)	Torque (N-m)
222.4	0.3398
333.6	0.3398
444.8	0.4077



Summary remarks

Based on the results obtained in this effort, the following conclusions are drawn:

- 1. The low wear and moderate friction coefficients suggest that PS400 is capable of providing solid lubricant protection for low sliding speed and high-temperature applications. The results confirm previous findings that the coating can produce the lubricious black oxide coating on surfaces that are in constant contact.
- 2. The dimensional instability experienced at 927°C indicates that the upper limit to PS400's service temperature falls between 760 and 927°C.
- 3. The bushing's surface finish after the EDM process was rougher than what would be commonly used in practice. It is expected that a smoother surface finish would have positively influenced the results.
- 4. The coating's predisposition for high temperatures was demonstrated by the excessive wear that occurred at 260°C due to dormancy of the fluorides and the low concentration of silver.
- 5. No signs of interstitial failure were observed, suggesting that the coating is capable of withstanding higher contact stress conditions. Additional testing is needed to determine this upper limit.

References

- (1) Miyoshi, K. (2007), "Solid Lubricants and Coatings for Extreme Environments: State-of-the-Art Survey," NASA/TM-2007-
- (2) Sliney, H. E. (1974), "Plasma-Sprayed Metal-Glass and Metal-Glass Fluoride Coatings for Lubrication to 900°C," ASLE Transactions, 17 (3), pp 182–189.
- (3) DellaCorte, C. (1988), "Tribological Composition Optimization of Chromium Carbide-Based Solid Lubricant Coatings for Foil Gas

- Bearings at Temperatures to 650°C," Surface and Coatings Technology, 36, pp 87-97.
- (4) DellaCorte, C. and Edmonds, B. J. (1995), "Preliminary Evaluation of PS300: A New Self-Lubricating High Temperature Composite Coating for Use to 800°C," NASA/TM-107056.
- (5) DellaCorte, C., Valco, M. J., Radil, K. C., and Heshmat, H. (2000), "Performance and Durability of High Temperature Foil Air Bearings for Oil-Free Turbomachinery," NASA/TM-2000-209187.
- (6) DellaCorte, C., Edmonds, B. J., and Benoy, P. A. (2001), "Thermal Processing Effects on the Adhesive Strength of PS304 High Temperature Solid Lubricant Coatings," NASA/TM-2001-210944.
- (7) Lubell, D., DellaCorte, C., and Stanford, M. (2006), "Test Evolution and Oil-Free Engine Experience of a High Temperature Foil Air Bearing Coating," Proceedings of GT2006: ASME Turbo Expo 2006, Barcelona, Spain, May 8-11.
- (8) DellaCorte, C. and Edmonds, B. J. (2009), "NASA PS400: A New High Temperature Solid Lubricant Coating for High Temperature Wear Applications," NASA/TM 2009-215678.
- (9) Blanchet, T. A., Kim, J.-H., Calabrese, S. J., and DellaCorte, C. (2002), "Thrust-Washer Evaluation of Self-Lubricating PS304 Composite Coatings in High Temperature Sliding Contact," Tribology Transactions, 45(4), pp 491-498.
- Striebing, D., Stanford, M. K., and DellaCorte, C. (2007), "Tribological Performance of PM300 Solid Lubricant Bushings for High Temperature Applications," NASA/TM-2007-214819.
- (11) Radil, K. C. and DellaCorte, C. (2009), "Foil Bearing Starting Considerations and Requirements for Rotorcraft Engine Applications," ARL-TR-4873.
- (12) National Aeronautics and Space Administration. (Sept. 2009) Available at: http://www.grc.nasa.gov/WWW/StructuresMaterials/Trib Mech/highlights/pm304.html (accessed January 2016).
- (13) Stanford, M. (2008), "Microstructure, Physical Properties, and Tribological Characteristics of Composite Solid Lubricant Coatings with Gas Atomized BaF2-CaF2," Tribology Transactions, 51(6), pp 829-
- (14) Stanford, M., Thomas, D. K., and DellaCorte, C. (2012), "Mechanical and Physical Properties of a PM Composite Solid-Lubricant Bushing," International Journal of Powder Metallurgy, 48(3), pp 41-49.

- 1 DEFENSE TECHNICAL
- (PDF) INFORMATION CTR DTIC OCA
 - 2 DIRECTOR
- (PDF) US ARMY RESEARCH LAB RDRL CIO L IMAL HRA MAIL & RECORDS MGMT
 - 1 GOVT PRINTG OFC
- (PDF) A MALHOTRA
- 22 DIR USARL (2 PDF, RDRL VTP
- 20 HC) K RADIL (1 PDF, 20 HC) C DELLACORTE

INTENTIONALLY LEFT BLANK.